DERWENT-ACC-NO: 1993-405562
DERWENT-WEEK: 199350

TITLE: Coating for machine tools - comprises nickel@, aluminium@, vanadium@,
oxygen and nitrogen, and is obtd. by three electrode cathodic sputter process
on static jig with biassing electrode to remove impurities
INVENTOR: BLESSING, D A
PATENT-ASSIGNEE: SAC INT INC[SACIN]
PRIORITY-DATA: 1992US-0893261 (June 4, 1992)
PATENT-FAMILY:

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DESIGNATED-STATES: CA JP KP KR RU UA US AT BE CH DE DK ES FR GB GR IE IT LU MC NL PT SE
CITED-DOCUMENTS: US 4419202; US 4871434 ; US 4973388
APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO	APPL-DATE
WO 9324316A1	N/A	1993WO-US05269	June 4, 1993

INT-CL_(IPC): B32B009/00; B32B019/00 ; C23C014/34
ABSTRACTED-PUB-NO: WO 9324316A
BASIC-ABSTRACT: The coating on a surface-coated article consists of at least Ti, Al, V, O and N. Also claimed is a process of coating a substrate by placing it proximate to an anode in a housing also including a cathodic target mfd. from at least Ti, Al and V, evacuating the housing to 0.1 to 0.001 torr and then introducing O2 and N2 while imposing a potential between target and anode to form the coating.

The coating also includes Mo and Y and is 2.0-8.0 x 10 power minus 5 and esp. 6.5 x 10 power minus 5 thick. Ti, Al and V, opt. Mo, are present in a nitride or oxide form. The target comprises Ti, Al, Mo and V and the potential between it and the anode is 500-1000 V. A bias electrode is included and has a potential relative to the cathode of 22.0-23.0 below the potential between anode and cathode. The vacuum is maintained at 0.01 torr and the gases are introduced in the wt. ratio 1-1.25 pt. O2 to 1 pt. N2. The cathode comprises concentric rings with the substrate located between them.

USE/ADVANTAGE - The substrate is a machine tool mfd. from steel, stainless
steel, titanium, carbide or inconel alloys and is uniformly coated over its
surface, or the substrate is Al_2O_3, Si_3N_4 or SiO_2. Other typical substrates
are airfoils and turbine blades. In a three electrode cathodic sputtering
process, a durable Ti-contg. coating is obtd. using a stationary jig and such
that the presence of a biasing electrode reduces inclusion of impurities in the
coating.
CHOSEN-DRAWING: Dwg.1/3
DERWENT-CLASS: L02 M13 P73 X25
CPI-CODES: L02-F02; L02-F03; L02-J01C; M13-G01;
EPI-CODES: X25-A04;

---------- KWIC ---------

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N. Also claimed is a process of coating a substrate by placing it proximate to
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coating.

TTX:
COATING MACHINE TOOL COMPRISE NICKEL@ ALUMINIUM@ VANADIUM@ OXYGEN
NITROGEN
OBTAIN THREE ELECTRODE CATHODE SPUTTER PROCESS STATIC JIG BIAS
ELECTRODE REMOVE
IMPURE

US-PAT-NO: 3732158
DOCUMENT-IDENTIFIER: US 3732158 A

TITLE: OCR SCANNED DOCUMENT
DATE-ISSUED: May 8, 1973

US-CL-CURRENT: 204/298.05,204/298.06 ,427/523 ,427/569

May 8, 1973 J. S. PRZYBYSZEWSKI ET AL 39732,158 METHOD AND APPARATUS FOR
SPUTTERING UTILIZING AN APERTURED ELECTRODE AND A PULSED SUBSTRATE BIAS Filed
Jan. 14 1971 0 MATCHIN 18 ORK RADIO FREQUENC POWER SOURCE 32 16 12 28 34 T 30
10 2 2 HIGH OLTAGE D.C. (0-5 KV.) 24 1 4 INVENTORS JOHN S. PRZYBYSZEWSKI
RICHARD K. SHALTENS BY ATTORNEYS
United States Patent Office 3973211 58 3,732,158 METHOD AND APPARATUS FOR
SPUTTERING UTILIZING AN APERTURED ELECTRODE AND A PULSED SUBSTRATE BIAS John S.
Przybyszewski, North Olmsted, and Richard K. 5 Shaltens, Lakewood, Ohio,
assignors to the United States of America as represented by the Administrator
of the National Aeronautics and Space Administration Filed Jan. 14, 1971, Ser.
No. 106,424 Int. Cl. C23c 15100 10 U.S. Cl. 204-192 7 Claims ABSTRACT OF THE
DISCLOSURE Combinin-g the advantages of ion plating with the ver-satility of a
radio frequency sputtered source. A pulsed high 1,5 voltage direct current is
passed to the article being plated during radio frequency sputtering. ORIGIN
OF THE INVENTION 20 The invention described herein was made by employees of the
United States Government and may be manufactured and used by or for the.
Govemment for governmental purposes without the payment of any royalties
thereon or 25 therefor. BACKGROUND OF THE INVENTION This invention is concemed
with plating adherent fflms p articularly dijrected to ion plating alloy films
on such o bjects using a radio frequency sputtered source. The ion p lating
process is modified because this source supplies fdm material at a much slower
rate than the usual thermal e vaporation source. 3 5 I n the past several
methods were used to deposit various t ypes of films on simple as well @as
geometrically complex o bjects. While each process is satisfactory for

certain a ppl,ications, problems have been encountered with all o f these methods. Conve,ntional vapor deposition is conducted 'm a vacu- U M of 10-6 to 10-8 torr. The use of conventional vapor d eposition has been generally restrieted to the, elemental metals, although some metal alloy syste@ms as well as cer- t ain semiconductors and nonconductors have been vapor d eposited. The high vacuum used in vapor depositio-n re- d uces the concentration of gas molecules which increases t he mean free path. Very little scattering of the film mate- ri a,l results, and the process is limited to line-of- sight d eposition. The coating of complex geometries by vapor d f,position is conditioned ion the rotatian of the object to b e coated. Adherence of a vapor deposited film is poor b ecause of the low energy of the impinging film material. The film adhesion is improved when either direct cur- r entOTradic frequency sputtering is used. D@irect current s puttering has been successful for depositing elemental metals, semiconductors, and metal alloy systems. This type o f sputtering is not useful for depositing nonconductors. Radio frequency sputtering has been used for depositing e lemental metals, metal alloy systems, semiconductors, a nd nonconductors. This type of sputteling is not limited b y the nature of the film material. It can be used to sput- t er almost any material from insulators through semi- con,ductors to metals. Radio frequency and direct current sputtering are generally done in an atmosphere having a pressure in the range of about 5 to 20 microns. Because of this relatively high pressure the sputtered material is scattered. The mean free path is short, and the material is diffused rapidly as it leaves the source. Even though this tscattering effect causes film formation on surfaces not directly facing the source P a t e n t e d M a y 8 , 1 9 7 3 2 material, both RF and DC sputtering are considered to be line-of- sight deposition processes. The low energy of the impinging film material adversely affects film adherence. Ion pl@ating is performed at about the same pressure as RF and DC sputtering. A high voltage is @applied to the object to be coated. This results in a uniform coating on all sides without rotating or moving

either the
object or the source of film material. WHe the coating has excellent adhesion,
problems have been encountered because, the process utilizes a thermial evaporation source. This lin-tits the filin materials to the elemental metals
and those compounds which do not dissociatebefore they evaporate. S U M M A R Y , O F T H E I N V - E N T I O N These pr(3blems have been solved by the
present in@vention which utilizes radio frequency sputtering with a pulse-d
high voltage direct current. The process is not liniited to a line-of-sight
deposition, and complex geometries can be plated without rotation. The process
is useful for plating adherent films of elemental metals, metal alloy systems,
semiconductors, and nonconductors. O B J . E C T S - O F T H E I N V E N T I O N It is, therefore, an object of the present invention to plate an adherent
alloy film on an object having a geometrically complex configuration. Another
object of the invention is to provide an improved plating method which combines
the advantages of tered source. A further object of the invention is to
provide an improved method for plating alloy filn-is on complex -eometries
without rotation during the plating process. These and other objects of the
invention will be apparent from the specification which follows and from the
drawing wherein hke numerals are used throughout to identify like parts.
DESCRIPTION OF THE DRAWING The figure is a schematic diagram of a system
constructed in accordance with the invention for plating adherent alloyed films
on geometrically complex objects. DESCRIPTION OF THE PREFERRED EMBODIMENT
Referring now to the drawing there is shown an object 10 which is to be coated
in accordance with the present invention. The object 10 may be any electrically conductive article having either a isimple or geometrically
complex configuration. By way of example the invention has been utilized to
coat bearings with ia solid lubricant. The object 10 is mounted in a chamber
12 that is connected at 14 to a suitable vacuum pumping system. A target 16 of
the material to bff, sputtered is likewise located in the chamber 12. The
target 16 is connected to a radio frequency power source 18 through;a matching

network 20. This RF sputtered source 16 is utilized instead of a thermal evaporation sourcenormally used in ion plating. As stated earlier, certain modifications are required because the RF sputtered source 16 supplies fitm material ata much slower rate than a thermal evaporation source. A suitable gas is supplied to the chamber 12 at an inlet 22. Argon is preferably used.
The object 10 to be plated is connected to a high voltage, dirert cutrent source 24. The -source 24- preferably has a range of 0 to 5 kilovolts. In this manner the ion plating is carried cut in a low pressure ionized gaseous atmosphere with the object 10 to be plated forming a cathode that is maintained at a high negative potential from the source 24. Consequently, the object 10 to be plated is continuously I>0mbarded or sputtered by -ions before, during and 'after film material on objects having complex geometries. The invention is 'o 0 ion plating with the versatility of a radio frequency sput-
3 enters the ionized gas. If the evaporation rate of the film material is too slow the film on the object to be plated will be sputtered away as fast as it develops, and no fflm will result. Because the RF sputtered source 16 is inherently slow, no film would develop under normal ion plating conditions.
According to the present invention the ion plating process has been modified to reduce the rate of sputtering off of the newly formed film by providing a timed switch 26 between the source 24 and the object 10. The reduction in the sputtering off rate is accomplished by pulsing the negative high voltage DC from the source 24 to the object 10 by means of the timed switch 26. A third electrode 28 is positioned in the chamber 12 to establish a common electrode between the RF power source 18 and the DC power source 24. The electrode 28 is connected to the high voltage DC power source 24 through an RF choke 30. The electrode 28 forms an anode with respect to the cathode 10. The radic frequency power source 18 is connected to the electrode 28 through a capacitor 32. A bypass capacitor 34 is likewise provided. This third electrode 28 is preferably in the form of a perforated plate or screen that is located between the sputtered source 16 and the object 10. The screen has an aperture

in the center to enable sputtered material to pass tO the object to be plated. In operation, the object 10 is mounted in the chamber 12 together with the target 16 of material to be sputtered. The chamber 12 is partially evacuated and a gaseous atmosphere from about 10-20 microns pressure is established. A high voltage DC negative potential of 2 to 5 kilovolts with respect to the screen 28 is continuously applied to the object 10. This establishes a glow discharge within the vacuum chamber 12 to sputter clean the object 10. After a predetermined period of sputter cleaning, the high voltage DC source 24 is deenergized and the gaseous pressure is lowered to about 1,0 microns. The RF power source 18 to the film material 16 is energized, and sputtering of the film material begins. At this point the high voltage DC source 24 is switched to a timed on-off mode by the switch 26. In this manner the high voltage DC is reenergized and reapplied to the object 10. The pulsed high voltage direct current RF sputtering process produces an intense electric field which completely surrounds the object 10. This can be seen as a dark space around this object. Any ionized material entering this region gains kinetic energy from the field and impacts on the surface of the object 10 with great force. This contributes to improved adhesion. The dark space, in effect, represents essentially a source of film material takes on the general outline of the object 10. The high voltage DC negative potential on the object 10 is maintained throughout the plating. The process is continued until the desired fUm is obtained. The reduced sputtering rate of the film on the object 10 results in the formation of a visible film having excellent adherence and covering the entire object. EY,AMPLE Pulsed hi_eh voltage direct current radio frequency sputtering was used to plate antifriction bearing components with a solid lubricant film of molybdenum disulfide. The plating conditions are as follows: Total coating time each component: 3 hours Radio Frequency Input Power to @ource Material: 700 watts at 7 megahertz Maximum pulse amplitude to specimen- 2000 V-DC, negative Pulse form: 15 seconds on; 5 minutes off; 5% duty cycle Specimen to screen distance: 2.5 inches 317321158 4 Specimen to source

distance: approximately 6 inches Chamber pressure: 5 microns; argon The components were assembled and the bearing was tested. The bearing was satisfactory for its intended use. While one embo,diment of the invention has been shown and described it will be apperciated that various modifications to the invention may be made without departing from the spirit of the invention or the scope of the subjoined claims. What is claimed is: 1. In a sputtering apparatus, including a vacuum chamber, means for admitting a gas into said chamber, target holding means for supporting the material to be sputtered, substrate holding means for supporting a substrate to be coated, means for applying RF potential to said target to sputter said material, and means for applying a high voltage direct current to said substrate; the improvement wherein an apertured electrode is disposed between said substrate holding means and said target holding means, said electrode being connected to said means for applying RF potential and said means for applying a high voltage direct current whereby a high voltage direct current negative potential with respect to said electrode is applied to said substrate holding means and an RF potential is applied to said target holding means to sputter said material, and timed switch means connected to said means for applying a high voltage direct current, said timed switch means enabling said high voltage direct current negative potential to be pulsed from of about 15 seconds on and about 5 minutes off. 2. Apparatus as claimed in claim I wherein said aper- tured electrode is a screen. 3. Apparatus as claimed in claim 2 wherein the screen has a centrally disposed aperture therein. 4. In an RF sputtering process wherein an RF potential is applied to a target to sputter material from the target onto a substrate, the improvement comprising disposing an apertured electrode between the target and the sub- strate, connecting said electrode to a source of RF potential and to a source of high voltage direct current, and applying to said substrate a high voltage direct current ne ative potential pulse from of about 15 seconds on and 9 about 5 minutes off. S. A method as claimed in claim 4 wherein said pulsed voltage is a potential of about 2 to 5 kilovolts. 6. A method as claimed in claim 4 including disposing

said target about 2.5 inches from said apertured electrode and said substrate about 6 inches from said apertured electrode. 7. A method as claimed in claim 6 wherein argon is utilized at a pressure of about 10 microns.

References Cited UNITED STATES PATENTS 3,361,659 1/1968 Bertelsen ----------- 204-298 3,526,584 9/1970 Shaw -------------- 204-298 3,528,906 9/1970 Cash et al ----------- 204-295 3,461,054 8/1969 Uratny ------------- 204-192 3,479,269 11/1969 Byrnes et al - -------- 204-192 3,530,055 9/1970 Maissel et al --------- 204-192 3,589,994 6/1971 Schwartz et al - ------ 204-192

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